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METHODS FOR SAMPLING OF MINERAL GYPSUM

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 4 *July* 1961

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Indian Standard

METHODS FOR SAMPLING OF MINERAL GYPSUM

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Indian Standard

METHODS FOR SAMPLING OF MINERAL GYPSUM

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 22 December 1960, after the draft finalized by the Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 Gypsum is a mineral of great importance in the industrial economy of the country. But as available, it is highly variable in quality and the percentage of calcium sulphate in any single deposit varies considerably.

0.2.1 Besides, the quality of gypsum also depends on the size of the particles, the larger particles generally having higher percentage of calcium sulphate. The average difference in calcium sulphate content between lumps (above 5 cm) and fines (5 cm and less) has been found from the investigations to vary considerably and is generally of the order of 6 percent. Investigations have further shown that it is due to more friable material containing silica which crumble, leaving lumps (above 5 cm) more concentrated in calcium sulphate.

0.2.2 It is, therefore, imperative that the lumps and fines be duly represented in the sample and the sampling procedure recommended in this standard is intended to ensure the same.

0.3 In the commercial sampling of gypsum, the usual procedure for drawing samples for test is to collect only one gross sample from a lot irrespective of its weight and test the same after reduction. The test result in this case obviously indicates only the average level of quality and does not give any indication of the variation in quality, knowledge of which is essential for proper quality evaluation of the material. The Sectional Committee responsible for the preparation of this standard, therefore, felt that for evaluating an important characteristic like calcium sulphate percentage it would be desirable to draw a number of gross samples instead of only one from a lot and test them separately after reduction. For the other characteristics, however, a composite sample prepared from the different gross samples could be tested.

0.3.1 Accordingly, this standard recommends the division of a lot into a number of sub-lots (*see* Table I) depending upon the tonnage and then drawing a representative gross sample from each of the sub-lots separately. The gross samples after suitable reduction are to be tested individually for important characteristics and compositely for others. The standard also lays down a proper procedure for valid interpretation of the test results in case of important characteristics, so as to evaluate the average level of quality and the extent of variation of the material in the lot. Finally, it indicates as to how a knowledge of the average and variation can be utilized in estimating the percentage of material above or below a certain quality level.

0.4 The recommendations made in this standard are primarily meant for determining the percentages of calcium sulphate and silica which form the basis for many trade transactions and which being variable, proper methods of sampling have to be adopted. But the recommendations are also expected to be adequate for the determination of other characteristics of gypsum, such as those specified in IS: 1290-1960 Specification for Mineral Gypsum for Ammonium Sulphate and Cement Industries.

0.5 Careful consideration was given to the various conditions under which gypsum may be available or supplied, for example, mineral reserves, stacks, wagons, conveyer belts, packages, etc. However, taking into account the many variations in the sampling conditions as also the commercial point of view, the Committee responsible for the preparation of this standard felt that the sampling procedures should be prescribed only for wagons, conveyer belts and packages.

0.6 In preparing this standard, considerable assistance has been derived from the experimental investigations carried out at Jamsar gypsum mines (near Bikaner) in Rajasthan by a working group consisting of the representatives of (a) Bikaner Gypsums Limited, (b) Sindri Fertilizers and Chemicals Private Ltd., (c) Associated Cement Companies of India, (d) General Superintendence Co. (India) Private Ltd., and (e) Indian Standards Institution.

0.7 Wherever a reference to any Indian Standard appears in this standard, it shall be taken as a reference to the latest version of the standard.

0.8 For the determination of the proportion of lumps and fines as well as for the reduction purposes, sieves conforming to IS: 460-1953 Specification for Test Sieves, shall be used. Where these sieves are not available, other equivalent standard sieves as indicated in IS: 460-1953 may be used.

0.9 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS: 2-1960 Rules for Rounding Off Numerical Values (*Revised*).

0.10 This standard is intended chiefly to cover the technical provisions relating to the sampling of gypsum, and it does not include all the necessary provisions of a contract.

1. SCOPE

1.1 This standard prescribes methods for sampling of gypsum from wagons, conveyer belts and packages. It also lays down a procedure for reporting the quality of the material sampled; and indicates the method for estimating the percentage of material above or below a certain level of quality.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

2.1 Lot —The quantity of gypsum delivered at one time in a shift or a day.

2.2 Sub-Lot — The quantity of gypsum in each of the portions into which a lot is divided for the purpose of sampling. A lot may be divided into several sub-lots.

2.3 Increment — The quantity of gypsum taken by a single operation of the sampling implement.

2.4 Gross Sample — Sample as collected from a sub-lot, that is, the quantity of gypsum obtained by aggregating together all the increments drawn from the same sub-lot.

2.5 Sample Division — The process whereby a part of the sample is retained and the remainder rejected.

2.6 Sample Reduction — The process of crushing or grinding the sample to reduce the particle size and of mixing and sample dividing in successive stages.

2.7 Laboratory Sample (or Prepared Sample) — The quantity of gypsum obtained by reducing a gross sample by following a specified procedure (*see* **2.6**) for laboratory testing.

2.8 Composite Sample (for the Lot) — The quantity of gypsum obtained by mixing together proportionate quantities of gypsum from each of the laboratory samples representing the sub-lots into which the lot has been divided.

2.9 Lumps — Gypsum retained on IS Sieve 2″.

2.10 Fines — Gypsum which passes through IS Sieve 2″.

3. SAMPLING FROM WAGONS

3.1 Sub-Lot — For the purpose of sampling, all the wagons in a lot shall be divided into a suitable number of sub-lots, depending upon the tonnage, in accordance with the requirements of Table I. The sub-lots shall, as far as practicable, be of approximately equal weights.

NOTE — While forming the sub-lots, they may consist of the wagons which are loaded with material as far as possible from the same quarry or loading point.

3.1.1 A representative gross sample shall be drawn from each of the sub-lots and shall be kept separately. Thus, there will be as many gross samples as the number of sub-lots into which the lot has been divided. The samples shall be drawn when gypsum is in regular motion during the loading or unloading of the wagons by manual operations.

TABLE I NUMBER OF SUB-LOTS

(*Clause* 3.1)

WEIGHT OF THE LOT tonnes	NO. OF SUB-LOTS
Up to 50	2
51 ,, 100	3
101 ,, 300	4
301 ,, 800	5
801 ,, 1 300	6
1 301 ,, 2 000	7

3.2 Gross Sample — The gross sample shall be collected from a sub-lot by taking about eight increments (*see also* Note 1) from each of the wagons in the sub-lot at regular intervals at the time of loading or unloading of the wagons manually. The increments shall be collected from a wagon four times during the whole period of its loading or unloading.

NOTE 1 — According to the existing conditions, an increment will consist of a pan-load of material weighing about 40 kg. The material taken out from a wagon at a time may consist of two pans generally, but sometimes only one pan may be actually moving at the time of sampling and hence that alone shall be collected for sampling. The quantity of material taken out from a wagon may, therefore, be about 320 kg.

NOTE 2 — Care shall be taken to ensure that the loading takes place in a regular and natural manner.

3.2.1 The material collected from all the wagons in a sub-lot shall constitute a gross sample.

3.3 Reduction of Gross Sample — All the increments collected under **3.2** shall be screened through IS Sieve 2". The lumps and fines so separated shall be kept aside.

3.3.1 The lumps and fines shall then be weighed separately and their proportion by weight shall be determined.

3.3.2 The lumps after weighing shall be separately broken to 5 cm size and quartered so as to obtain about 70 kg of material.

The fines after weighing shall also be separately quartered so as to obtain about 70 kg of material.

3.3.3 The samples of lumps and fines as collected under **3.3.2** shall be crushed separately so as to pass through IS Sieve 320 and riffled till the quantity comes down to 10 kg in each case.

3.3.4 *Laboratory Sample (or Prepared Sample)* — Proportional weights of lumps and fines, as determined under **3.3.1**, shall be taken from the crushed material (*see* **3.3.3**) and after further crushing shall be thoroughly mixed together to pass IS Sieve 85. The sieved material shall be reduced by coning and quartering (*see* Appendix B) and crushed to pass through IS Sieve 25. It shall then be reduced further by coning and quartering till the quantity comes down to about 1·5 kg which shall constitute the laboratory sample for the sub-lot.

3.3.4.1 If a sample is required for the determination of free moisture, the material obtained by mixing the proportional weights of lumps and fines (*see* **3.3.4**) shall be crushed and thoroughly mixed together to pass IS Sieve 100 (instead of IS Sieve 85). A suitable quantity of the material, sufficient for the test, shall then be withdrawn before further crushing it to pass IS Sieve 25.

3.3.4.2 If due to excessive free moisture, it is difficult to sieve the material through IS Sieve 25, the material may be dried at a temperature of 45°± 1°C before reducing the material to IS Sieve 25 size.

3.3.4.3 Each laboratory sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee. The samples shall be kept in glass or polythene containers and shall be sealed and marked properly.

3.4 A lot shall be represented by two or more laboratory samples depending upon the number of sub-lots into which the lot has been divided.

3.5 To illustrate the procedure for the collection and reduction of gross sample, a schematic representation for a typical case is given in Fig. 1 and 2 respectively.

4. SAMPLING FROM CONVEYER BELTS

4.0 While sampling from a conveyer belt, the material should preferably be crushed to about 5 cm.

4.1 Sub-Lots — For the purpose of sampling, a lot while it is being discharged over a conveyer belt shall be divided into a number of sub-lots as specified in Table I.

4.1.1 A gross sample shall be drawn from each of the sub-lots and shall be kept separately. Thus, there will be as many gross samples as the number of sub-lots into which the lot has been divided.

4.2 Gross Sample — The gross sample shall be collected by taking four increments (*see also* Note below) from a sub-lot at regular intervals.

NOTE — The total quantity of the material taken out from a sub-lot shall be not less than 70 kg; additional increments may be suitably taken for this purpose, if necessary.

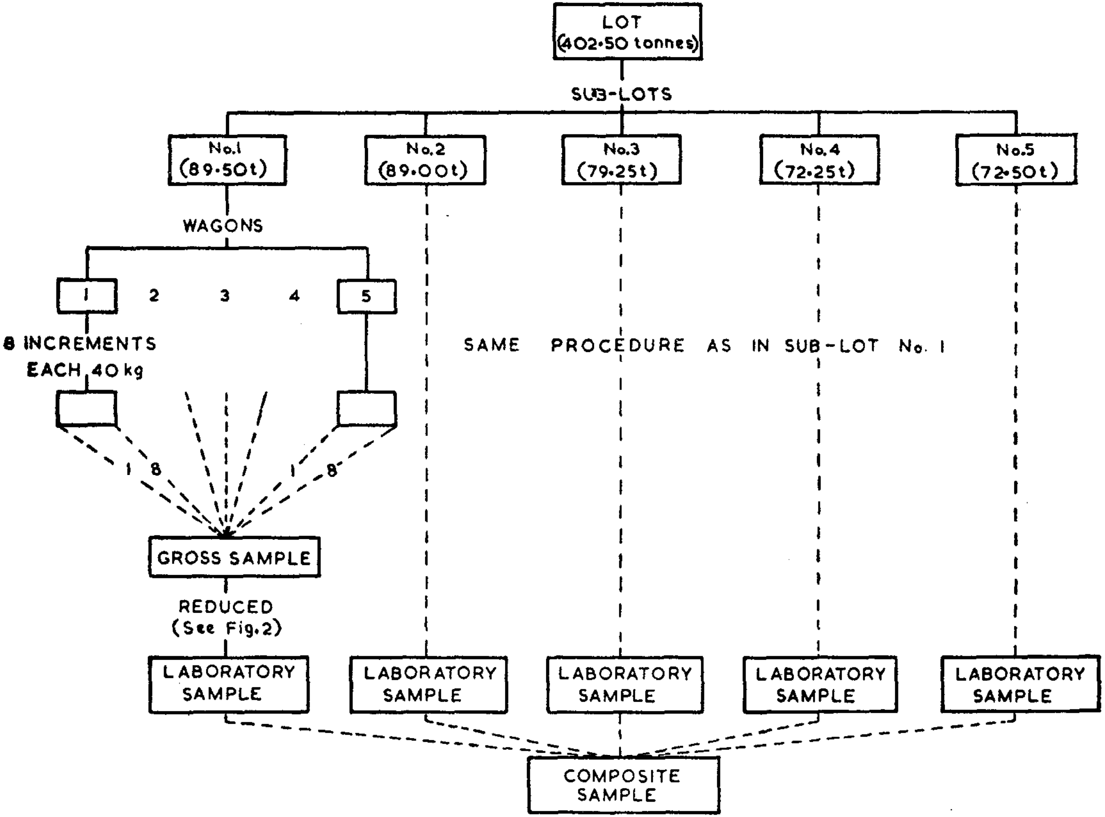

Fig. 1 Collection of Gross Sample and Composite Sample

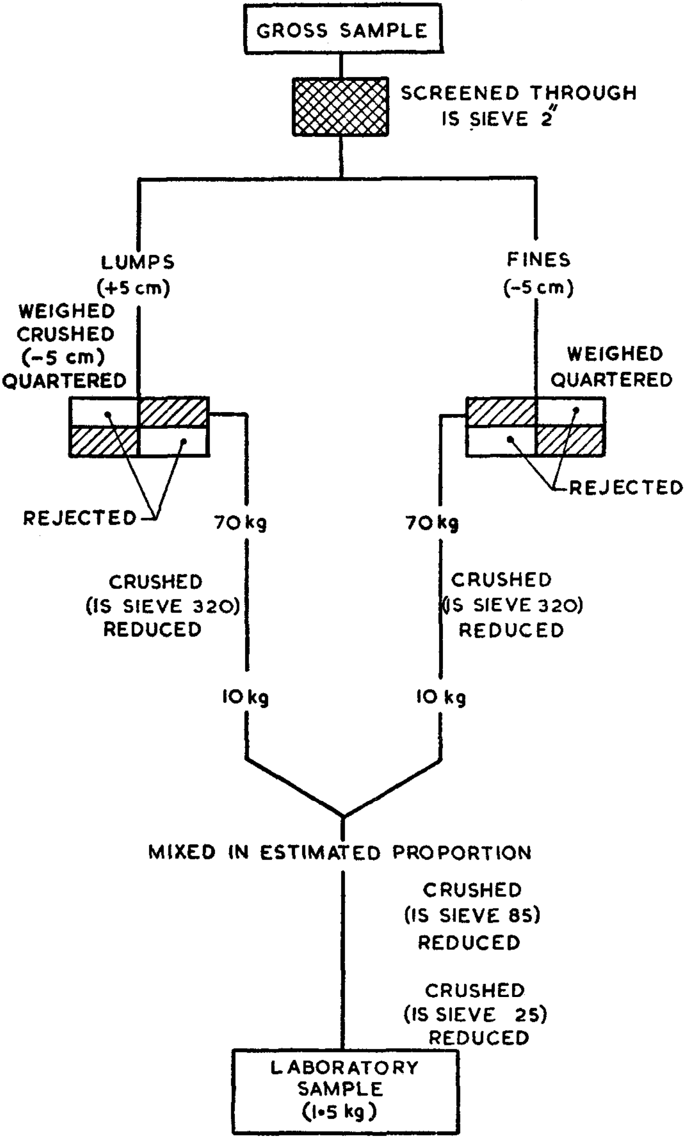
GROSS SAMPLE
SCREENED THROUGH IS SIEVE 2"
LUMPS (+5 cm)
FINES (-5 cm)
WEIGHED CRUSHED (-5 cm) QUARTERED
WEIGHED QUARTERED
REJECTED
REJECTED
70 kg
70 kg
CRUSHED (IS SIEVE 320) REDUCED
CRUSHED (IS SIEVE 320) REDUCED
10 kg
10 kg
MIXED IN ESTIMATED PROPORTION
CRUSHED (IS SIEVE 85) REDUCED
CRUSHED (IS SIEVE 25) REDUCED
LABORATORY SAMPLE (1·5 kg)

FIG. 2 REDUCTION OF GROSS SAMPLE

4.2.1 The increments shall preferably be taken from the full cross section and thickness of the stream and in one operation. When gypsum is in motion, the most reliable means of obtaining such increments is to sample at a point where the gypsum discharges from the belt. The best possible increment is one which cuts across entirely a falling stream of the material by means of a suitable receptacle passed from one side of the stream to the other without allowing the receptacle to overflow. If the whole of the stream cannot be covered by one increment without overfilling the receptacle, the stream should be sampled systematically by taking material from all portions.

4.2.2 If it is not possible to sample satisfactorily at the point of discharge, the increments may be drawn from the moving belt itself. In this case, the increments shall be collected from the centre and the left and right sides of the belt along the same width. To ensure that very fine material is also correctly obtained in the sample, the scoop should sweep the bottom of the conveyer.

4.2.3 If it is practicable to stop the belt periodically, increments may be collected from the whole cross section of the stream by sweeping the whole of the gypsum lying between the sides of a suitable frame placed across the belt. The frame should be inserted in the gypsum until it is in contact with the belt across its full width.

NOTE — Before collecting the increments, the speed of the conveyer and quantity of material passing a certain point in a given time shall be ascertained so that an appropriate spacing of the increments may be arranged over the whole of the lot.

4.2.4 In case suitable automatic samplers are available, they may be utilized for drawing increments from a conveyer belt. The setting of such machines shall be carefully adjusted to ensure that the whole thickness of the stream is taken.

4.2.5 The material collected from all the increments in a sub-lot shall be mixed together and shall constitute a gross sample.

4.3 Reduction of Gross Sample — The gross sample drawn from a sub-lot shall be mixed together and crushed to pass IS Sieve 320 and riffled till the quantity comes down to 10 kg.

4.3.1 *Laboratory Sample* (*or Prepared Sample*) — The material as obtained under **4.3** shall be crushed and thoroughly mixed together to pass IS Sieve 85. The sieved material shall be reduced by coning and quartering and crushed to pass IS Sieve 25 (*see also* **3.3.4.1** and **3.3.4.2**). It shall then be reduced further by coning and quartering till the quantity comes down to about 1·5 kg which shall constitute the laboratory sample for the sub-lot. The laboratory sample shall then be divided into three parts as in **3.3.4.3**.

5. SAMPLING FROM PACKAGES (UNIFORM FINE MINERAL)

5.1 Sub-Lot — All the packages in a lot shall be divided into a suitable number of sub-lots, depending upon the tonnage, as specified in Table I.

5.1.1 A representative gross sample shall be drawn from each of the sub-lots as specified in **5.2**, and shall be kept separately for test purposes. Thus, there will be as many gross samples as the number of sub-lots into which the lot has been sub-divided.

5.2 Gross Sample — For drawing a representative sample from a sub-lot, at least three percent of the packages, with the minimum of five, shall be sampled. In case fractional numbers are obtained, the number of packages to be selected shall be taken to be equal to the next higher integer.

5.2.1 These packages shall be chosen at random from the sub-lot. To ensure such selection, a random number table as agreed between the purchaser and the supplier shall be used. In case such a table is not available, the following procedure shall be adopted:

> Arrange all the packages in the sub-lot in one order and starting from any package, count them as 1, 2, 3, ... , up to r and so on. Every rth container, thus counted, shall be separated to give a sample for test, where $r = M/m$, M being the total number of packages in the lot, and m the number of packages to be chosen (*see* **5.2**). In case r is not a whole number, its value shall be taken as equal to the integral part of it.

5.2.2 Draw with an appropriate sampling scoop small portions of the material from different parts of each package selected (*see* **5.2**). The total quantity of the material taken out from each package shall be not less than 5 kg.

5.2.2.1 The material drawn from various packages in a sub-lot shall be thoroughly mixed together and shall constitute a gross sample.

5.3 Laboratory Sample — The material as obtained under **5.2.2.1** shall be riffled till the quantity comes down to 10 kg. It shall then be further reduced and a laboratory sample prepared as in **4.3.1**.

6. NUMBER OF TESTS

6.1 The laboratory samples representing various sub-lots in a lot shall be tested individually for important characteristics. For the remaining characteristics, a composite sample prepared by mixing proportionate quantities by weight of the material from each of the laboratory samples shall be tested.

6.2 Unless otherwise agreed between the purchaser and the vendor or specified in the material specification, the characteristics for which the individual and the composite samples are to be tested shall be in accordance with Table II.

TABLE II SAMPLE FOR TESTS

(*Clause* 6.2)

CHARACTERISTIC	SAMPLE
i) Calcium sulphate ($CaSO_4.2H_2O$)	Individual
ii) Silica (for ammonium sulphate industry only)	
iii) Silica (for industries other than ammonium sulphate industry)	Composite
iv) Iron and aluminium (as oxides)	
v) Magnesium oxide (MgO)	
vi) Chlorides (as NaCl)	

7. REPORTING OF TEST RESULTS

7.1 For those characteristics where a composite sample has been tested, only one test result shall be available and that result shall be reported as the value of the characteristic for the lot.

7.2 In the case of calcium sulphate and silica where the laboratory samples have been tested individually, the weighted average ($\bar{x}$) and the standard deviation (s) of the test results shall be calculated (*see* **A-1**) for assessing the average level and the extent of variation of the quality.

7.2.1 The average level of the characteristic in the lot shall be reported as equal to $\bar{x}$.

7.2.2 The limits for the variation of the characteristic in the lot shall be reported as $\bar{x} \pm Ks$, where K is a constant, the value of which depends upon the number of laboratory samples analysed (*see* Table III), implying thereby that the quality of the material in the lot varies between these limits.

7.3 For assessing the percentage of material lying above or below a certain level of quality, the procedure prescribed in Appendix A shall be followed.

TABLE III VALUES OF THE CONSTANTS *K* AND *c*

(*Clauses* 7.2.2 *and* A-2.1)

NUMBER OF LABORATORY SAMPLES ANALYSED (n)	VALUE OF K	VALUE OF c
2	3·54	0·564
3	2·76	0·724
4	2·51	0·798
5	2·38	0·841
6	2·30	0·869
7	2·25	0·888

APPENDIX A

(*Clause* 7.3)

ASSESSMENT OF QUALITY

A-1. The weighted average and the standard deviation of the test results shall be calculated as follows:

If, for any particular characteristic, $x_1, x_2, \ldots, x_n$ are the results of analysing n laboratory samples representing the sub-lots of weights $w_1, w_2, \ldots, w_n$ respectively, then

$$\text{Weighted average } (\bar{x}) = \frac{w_1x_1+w_2x_2+\ldots+w_nx_n}{w_1+w_2+\ldots+w_n} = \frac{\Sigma wx}{\Sigma w}$$

$$\text{and standard deviation } (s) = \sqrt{\left\{\frac{w_1(x_1-\bar{x})^2+w_2(x_2-\bar{x})^2+\ldots+w_n(x_n-\bar{x})^2}{w_1+w_2+\ldots+w_n}\right\}}$$

$$= \sqrt{\left\{\frac{\Sigma w(x-\bar{x})^2}{\Sigma w}\right\}}$$

$$= \sqrt{\left\{\frac{\Sigma wx^2}{\Sigma w} - \bar{x}^2\right\}}$$

A-2. The values of the weighted average ($\bar{x}$) and the standard deviation (s) obtained in **A-1** may be utilized in assessing the percentage of material lying above or below a certain level of quality or between two levels of quality.

A-2.1 For estimating the percentage of material below a quality level, say L, the value of the expression $(L-\bar{x})/\sigma$ ($= t_L$ say) will be calculated, where $\sigma = s/c$ and c is read from Table III for the given n. The proportion below t_L, say P_L, which is read off from Table IV, determines the proportion of material having a quality level less than or equal to L.

A-2.1.1 $100P_L$ and WP_L shall give respectively the estimates of the percentage and the actual amount of material having a quality worse than or equal to L, W being the total weight of the lot.

A-2.2 The proportion of material having a quality level of U or more may be estimated by calculating the value of the expression $(U-\bar{x})/\sigma (= t_U$ say) and then taking the proportion beyond this value which shall be equal to $(1-P_U)$ where P_U is directly read off from Table IV as before.

A-2.2.1 $100(1-P_U)$ and $W(1-P_U)$ shall give respectively the estimates of percentage and the actual amount of material having a quality better than or equal to U.

TABLE IV PROPORTION OF MATERIAL BELOW *t*

(*Clauses* A-2.1, A-2.2 *and* A-3)

	PROPORTION OF MATERIAL (P)		PROPORTION OF MATERIAL (P)
−3·0	0·001 3	+0·0	0·500 0
−2·8	0·002 6	+0·1	0·539 8
−2·6	0·004 7	+0·2	0·579 3
−2·4	0·008 2	+0·3	0·617 9
−2·2	0·013 9	+0·4	0·655 4
		+0·5	0·691 5
−2·0	0·022 8	+0·6	0·725 7
−1·9	0·028 7	+0·7	0·758 0
−1·8	0·035 9	+0·8	0·788 1
−1·7	0·044 6	+0·9	0·815 9
−1·6	0·054 8	+1·0	0·841 3
−1·5	0·066 8	+1·1	0·864 3
−1·4	0·080 8	+1·2	0·884 9
−1·3	0·096 8	+1·3	0·903 2
−1·2	0·115 1	+1·4	0·919 2
−1·1	0·135 7	+1·5	0·933 2
−1·0	0·158 7	+1·6	0·945 2
−0·9	0·184 1	+1·7	0·955 4
−0·8	0·211 9	+1·8	0·964 1
−0·7	0·242 0	+1·9	0·971 3
−0·6	0·274 3	+2·0	0·977 3
−0·5	0·308 5		
−0·4	0·344 6	+2·2	0·986 1
−0·3	0·382 1	+2·4	0·991 8
−0·2	0·420 7	+2·6	0·995 3
−0·1	0·460 2	+2·8	0·997 4
−0·0	0·500 0	+3·0	0·998 7

NOTE — Linear interpolation may be used where necessary.

A-2.3 The percentage and the actual amount of material lying between the two quality limits L and U shall be given by $100(P_U - P_L)$ and $W(P_U - P_L)$ respectively.

A-3. ILLUSTRATIVE EXAMPLE

A-3.1 In a sampling investigation conducted to determine the quality of a lot of gypsum, five gross samples were collected and analysed for calcium sulphate percentage. The test results obtained were:

89·09, 86·02, 83·38, 88·69 and 91·76

representing respectively the tonnages of

89·50, 89·00, 79·25, 72·25 and 72·50.

It is required to estimate the percentage of material with sulphate content of (a) 86 percent or less and (b) 87 percent or more.

The weighted average ($\bar{x}$) and the standard deviation (s) of the five test results are respectively equal to 87·70 and 2·81. The value of $\sigma(= s/c)$ is calculated to be $\frac{2{\cdot}81}{0{\cdot}841} = 3{\cdot}34$

a) For estimating the percent material below 86, the value of the expression $(86-87{\cdot}70)/3{\cdot}34$ is found to be equal to $-0{\cdot}5$. Reading from Table IV corresponding to this value, P_L is found to be equal to 0·308 5 or 31 percent. This indicates that 31 percent of the material has sulphate content of 86 percent or less. As the total tonnage is 402·50 tonnes, the amount of material having a quality worse than 86 percent is 125 tonnes.

b) For estimating the percent material over 87 percent, the value of the expression $\left(\frac{87-87{\cdot}70}{3{\cdot}34}\right)$ is found to be equal to $-0{\cdot}2$.

Corresponding to this value, P_U is read from Table IV as equal to 0·420 or 42 percent. This implies that the sulphate content in 58 percent of the material is 87 percent or more. As the total tonnage is 402·50 tonnes, the amount of material having a quality better than 87 percent is 233 tonnes.

It follows from the above that the material having sulphate percentage between 86 and 87 is 11 percent or 44 tonnes.

APPENDIX B

(*Clause* 3.3.4)

PREPARATION OF LABORATORY SAMPLES

B-1. GENERAL PRECAUTIONS

B-1.1 The places set apart for the treatment of gross and laboratory samples shall preferably be enclosed, roofed over, cool and free from draughts.

B-1.2 Where this is not possible, precautions shall be taken against (a) loss of fine wind-borne sample, (b) contamination with moisture, and (c) contamination with foreign matter.

B-1.3 Select a hard and clean surface free of cracks for sample mixing, quartering and other operations. Do not let cinders, sand, chippings from the floor or any other foreign matter get into the sample.

B-1.4 While grinding the samples, care shall be taken to see that excessive heat is not generated so as to induce calcination (dehydration) of the samples.

B-2. CONING AND QUARTERING

B-2.1 The material which has passed through IS Sieve 85 (*see* **3.3.4**) shall be heaped into the shape of a cone by pouring one scoopful of the material after another at the apex of the cone till the entire sample has been coned. The material shall be allowed to slide down the sides of the cone only under the influence of gravity.

B-2.2 Flatten the cone evenly so that it forms a low circular pile. Cut the pile into four quarters along two diameters which intersect at right angles. Retain one pair of opposite quarters and reject the other. Repeat till the size of the retained sample is reduced to the required weight of 1·5 kg.

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